Data Evaluation Report on the adsorption-desorption of iodomethane in soil

PMRA Submission Number {......} EPA MRID Number 45593709

Data Requirement: PMRA Data Code:
EPA DP Barcode: D280800
OECD Data Point:
EPA Guideline: 163-1

Test material:
Common name: Iodomethane.
Chemical name
IUPAC: N/A.
CAS name: Iodomethane.
CAS No: 74-88-4.
Synonyms: Methyl iodide.
TM-425.
SMILES string: CI

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Active Code: [for PMRA]
Use Site Category: [for PMRA]
EPA PC Code: 000011

CITATION: McFadden, J.J. and C.R. Landphair. 2001. Adsorption and desorption of [^{14}C]iodomethane (TM-425) on five soils. Unpublished study performed by Ricerca, LLC, Concord, OH, and sponsored by Arvesta Corporation, San Francisco, CA. Document Number 013136-1. Project Identification Number 013136. Study initiated January 11, 2001 and completed November 27, 2001.

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EXECUTIVE SUMMARY:

The adsorption/desorption characteristics of [^{14}C]iodomethane [TM-425; iodomethane] was studied in a sandy loam soil [pH- 6.9, organic carbon - 1.02%], clay loam soil [pH - 7.9, organic carbon - 4.3%], silt loam soil [pH - 6.2, organic carbon - 1.95%], each from the U.S., a loam soil [pH- 7.5, organic carbon - 1.76%] from Switzerland, and a sandy loam soil [pH - 6.3, organic carbon - 2.73%] from Germany, in a batch equilibrium experiment. The soils were sterilized using gamma irradiation (≥26.1 kGy). The experiment was conducted in accordance with the U.S. EPA Pesticide Guidelines Subdivision N, 163-1 and OECD Guidelines for Testing of Chemicals, and in compliance with the GLP standard 40 CFR Part 160. The adsorption phase of the study was carried out by equilibrating soil with [^{14}C]iodomethane at nominal concentrations of 9.05, 2.9, 0.88 and 0.29 mg a.i./kg at 20°C for 24 hours (lighting conditions not reported). The equilibrating solution used was 0.01 M $CaCl_2$, with soil/solution ratios of 1:3 (w:v) for the five soils. The desorption phase of the study was carried out by replacing the adsorption solution with an equivalent volume of sterilized, pesticide-free 0.01 M $CaCl_2$ solution and equilibrating for 24 hours at 20°C. The desorption phase was conducted once.

The supernatant solution after adsorption and desorption was separated by centrifugation and duplicate aliquots (1.0 mL) were analyzed using LSC. Radioactivity in the soil residue after desorption was determined by combustion. Portions of soil were combusted and analyzed by LSC.

The mass balance at the end of adsorption phase of the study was not reported. The complete mass balance for the high-dose soils (adsorption and desorption phase) was 87.0-88.3%, 84.4-87.6%, 86.4-89.8%, 91.4-93.9%, and 86.7-90.4% of the applied for the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively.

After 24 hours of equilibration, 13.8-15.0%, 13.9-19.8%, 25.7-31.9%, 9.3-14.4%, and 19.3-25.1% of the applied [^{14}C]iodomethane was adsorbed to the loam, California sandy loam, clay loam, German sandy loam and silt loam soils, respectively (reviewer-calculated). Freundlich K_{ads} values were 0.5, 0.6, 1.1, 0.4, and 0.8 mL/g for the loam, California sandy loam, clay loam, German sandy loam and silt loam soils, respectively; corresponding Freundlich K_{oc} values were 28, 59, 26, 15, and 41.

At the end of desorption, 0%, 8.7-16.8%, 13.8-20.9%, 0-9.2%, and 15.6-25.0% of the applied ^{14}C was desorbed from the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively (reviewer-calculated). Freundlich K_{des} values were 2.5, 2.7, 2.9, 2.1, and 2.4 mL/g for the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively; corresponding Freundlich K_{oc} values were 142, 265, 67, 77, and 123.

Results Synopsis: Adsorption and desorption K values were determined by the study author using Freundlich isotherm equations. Amounts adsorbed and desorbed were reviewer-calculated.

Soil type: Loam
Amount adsorbed: 13.8-15.0% of the applied
Adsorption K_d: 0.5
Adsorption K_{oc}: 28
Amount desorbed: 0%
Desorption K_d: 2.5
Desorption K_{oc}: 142

Soil type: California sandy loam
Amount adsorbed: 13.9-19.8% of the applied
Adsorption K_d: 0.6
Adsorption K_{oc}: 59
Amount desorbed: 8.7-16.8% of the adsorbed
Desorption K_d: 2.7
Desorption K_{oc}: 265

Soil type: Clay loam
Amount adsorbed: 25.7-31.9% of the applied
Adsorption K_d: 1.1
Adsorption K_{oc}: 26
Amount desorbed: 13.8-20.9% of the adsorbed
Desorption K_d: 2.9
Desorption K_{oc}: 67

Soil type: German sandy loam
Amount adsorbed: 9.3-14.4% of the applied
Adsorption K_d: 0.4
Adsorption K_{oc}: 15
Amount desorbed: 0-9.2% of the adsorbed
Desorption $K_{d:}$ 2.1
Desorption K_{oc}: 77

Soil type: Silt loam
Amount adsorbed: 19.3-25.1% of the applied
Adsorption K_d: 0.8
Adsorption K_{oc}: 41
Amount desorbed: 15.6-25.0% of the adsorbed
Desorption $K_{d:}$ 2.4

Desorption K_{oc}: 123

Study Acceptability: This study is classified as **supplemental**. Although the study was performed according to Subdivision N Guideline, insufficient information on experimental design was provided to verify the conclusions.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: The study was conducted according to U.S. EPA Pesticide Assessment Guidelines Subdivision N, Series 163-1, the Organization for Economic and Cooperative Development (OECD), and Guideline for Testing of Chemicals, "Adsorption/Desorption", Guideline 106 (p. 17). Although no significant deviations from Subdivision N Guideline §163-1 were identified, inadequate information (experimental design and HPLC analytical methods) was provided about the study design and analytical methods to determine if the study is scientifically valid and meets guideline requirements.

COMPLIANCE: This study was conducted in compliance with 40 CFR Part 160, EPA GLP. Signed and dated GLP, Quality Assurance, Data Confidentiality, and Study Certification statements were provided (pp. 2, 3, 6, and 7).

A. MATERIALS:

1. Test Material $[^{14}C]$Iodomethane

Chemical Structure: H_3C^*-I (* location of radiolabel).

Description: Not provided

Purity: Analytical purity: Not provided
Lot/Batch No.: 110K 9406/07
Radiochemical purity: ≥95% (p. 18)
Specific activity: 6.1 mCi/mmol
Location of the label: Methyl carbon

Storage conditions of test chemicals: Not provided

Physico-chemical properties of iodomethane:

Parameter	**Values**	**Comments**
Molecular weight	141.94 g/Mol	
Water solubility	14.2 mg/mL at 25°C	
Specific gravity	2.8 at 20°C	Material Safety Data Sheet
Vapor pressure	400 mm/Hg at 25°C 50 kPa at 20°C	Material Safety Data Sheet International Occupational Safety and Health Information Centre
Henry's law K_H	0.22 dimensionless	Estimated
UV absorption	Maximum (2.5 absorbance units) at *ca.* 200 nm, with a smaller peak (0.25 au) at *ca.* 250 nm	MRID 45593706
pK_a	Not applicable	
Octanol/Water partition coefficient (log K_{ow})	1.51-1.69	International Occupational Safety and Health Information Centre
Melting point	-66.5°C	International Occupational Safety and Health Information Centre
Boiling point	42.4°C	
Stability of compound at room temperature, if provided	Not reported	

Data obtained from p. 18 of the study report unless otherwise noted.

2. Soil Characteristics

Table 1: Description of soil collection and storage.

Description	Sandy loam	Sandy loam	Clay loam	Loam	Silt loam
Geographic location	Speyer, Germany	Watsonville, CA	Painsville Township, OH	Switzerland	Hillsboro, OR
Pesticide use history at the collection site	Not provided	Not provided	Not provided	Not provided	Not provided
Collection procedures	Not provided	Not provided	Not provided	Not provided	Not provided
Sampling depth (cm)	Not provided	Not provided	Not provided	Not provided	Not provided
Storage conditions	Not provided	Not provided	Not provided	Not provided	Not provided
Storage length	Not provided	Not provided	Not provided	Not provided	Not provided
Soil preparation	Sieved, 2 mm	Sieved, 2 mm	Sieved, 2 mm	Sieved, 2 mm	Sieved, 2 mm

Data were obtained from p. 20 and Table 1, p. 45 of the study report.

Table 2: Properties of the soils.

Property	Swiss	California	Ohio	German	Oregon
Soil Texture	Loam	Sandy loam	Clay loam	Sandy loam	Silt loam
% sand	43.2	74.8	42	73.2	19.2
% silt	37.6	15.6	30	17.6	59.6
% clay	19.2	9.6	28	9.2	21.2
pH	7.5	6.9	7.9	6.3	6.2
Organic carbon (%)	1.76	1.02	4.3	2.73	1.95
Organic matter (%)	3.03	1.76	7.4	4.70	3.35
CEC (meq/100 g)	10.43	9.72	18.1	6.65	11.18
Moisture at 1/3 atm (%)	20.53	10.99	34.4	15.35	26.05
Bulk density (g/cm^3)	1.18	1.51	1.02	1.21	1.41
Biomass (mg microbial C/100 g or CFU or other)	Not provided	Not provided	Not provided	Not provided	Not provided
Soil taxonomic classification	NA	Cumulic Haploxeroll	Aeric Ochraqualf	Mollisol	Typic Hapludand
Soil mapping unit (for EPA)	Not provided	Not provided	Not provided	Not provided	Not provided

Data were obtained from Table 1, p. 45 of the study report.

B. STUDY DESIGN:

1. Preliminary study: To determine the soil:solution ratio to be used in the definitive study, samples were prepared by adding 7, 6 or 5 mL of 0.01 M $CaCl_2$ solution to vessels containing 1, 2, or 4 g of sandy loam or clay loam soil (dry weight equivalent), resulting in soil:solution ratios of 1:7, 1:3, and 1.25 (w:v), respectively (pp. 21-22). The soils were pre-equilibrated overnight by shaking at 20°C. An aliquot (16-22 µL) of [^{14}C]iodomethane was added at a nominal concentration of 0.1 µg/mL (Table 2, p. 46). The vessels were shaken at approximately 20°C for about 24 hours, then the samples were centrifuged. Triplicate aliquots (1 mL) of the supernatant were analyzed for total radioactivity using LSC. An aliquot (1 mL) of the supernatant from each soil tested using the test tube from the 1:3 soil:solution ratio was analyzed by HPLC to determine the stability of [^{14}C]iodomethane in the $CaCl_2$ supernatant solution (p. 22).

To determine the potential for the test substance to adsorb to the glass screw cap centrifuge tubes, two test vessels containing 0.01 M $CaCl_2$ without soil were treated with approximately 0.1 μg/mL of [^{14}C]iodomethane and shaken on a reciprocal shaker for about 24 hours at 20°C. Aliquots were analyzed using LSC (p. 22). The results were compared to initial values to determine radiocarbon recovery and adsorption to glass.

Three preliminary adsorption kinetics experiments were conducted. The first experiment was conducted to estimate equilibration times using the California sandy loam soil and the Ohio clay loam soil, representing the range of clay content of the test soils to be used in the definitive study (p. 23). The test tubes were silanized with dimethyldichlorosilane to minimize adsorption of the test substance to the glass vessels. Fourteen samples of sandy loam and clay loam soils (2 g, dry weight equivalent) were pre-equilibrated overnight with 6 mL of a 0.01 M $CaCl_2$ solution by shaking on a reciprocal shaker at 20°C the day before the experiment (p. 23). The soils were then treated with 19 μL of a 0.01 M $CaCl_2$ solution containing [^{14}C]iodomethane; the final test substance concentration was 0.1 μg/mL. The samples were shaken on a reciprocal shaker for up to 72 hours at 20°C. Duplicate samples were removed after 2, 4, 6, 12, 24, 48, and 72 hours of shaking, then centrifuged at room temperature. Triplicate 0.1-mL aliquots were analyzed for total radioactivity using LSC. The adsorption equilibrium time was determined based on the percent adsorption of the test substance to each test soil. To determine the stability of [^{14}C]iodomethane in the soil:$CaCl_2$ supernatant solution, aliquots of supernatant samples collected after 24 hours of shaking were analyzed by HPLC. To determine the mass balance of the test substance, single samples of each soil were collected after 24 and 48 hours of shaking, and combusted and analyzed by LSC.

A second preliminary study was conducted using the California sandy loam soil to determine equilibration time and stability of iodomethane in sterilized and non-sterilized soil (p. 24). Portions of sterilized (autoclaved for 30 minutes at 121°C) and non-sterilized soil (2 g, dry weight equivalent) were added with 7.5 mL of 0.01 M $CaCl_2$ (1:3.75) to non-silanized glass test tubes. Three individual replicate tubes were prepared for each of the following sets of samples: non-sterilized soil treated at 0.1 μg/mL, sterilized soil treated at 0.1 μg/mL, and non-sterilized soil treated at 3 μg/mL. The test vessels were pre-equilibrated overnight on a reciprocal shaker at 20°C the day before the experiment. The tubes were removed after 1, 4, and 24 hours of shaking and centrifuged at room temperature. Triplicate 0.1-mL aliquots were analyzed for total radioactivity using LSC. The adsorption equilibration time was determined based on the percent adsorption of the test substance to each soil. To determine the stability of [^{14}C]iodomethane in the soil-$CaCl_2$ supernatant solution at the 1-, 4-, and 24-hour sampling intervals, aliquots (0.25 or 1 mL) of the supernatant samples were analyzed using HPLC. To determine the potential of the test substance to adsorb to the test vessels during the preliminary tests, $CaCl_2$ solutions containing approximately 0.1 or 1.35 μg/mL of [^{14}C]iodomethane were placed in silanized and non-silanized glass vessels without soil and shaken at 20°C for about 18 hours (p. 25). Triplicate aliquots (25 μL) were analyzed by LSC.

A third adsorption kinetics experiment was conducted using all five test soils to be used in the definitive study. To determine the level of microbial degradation under the test conditions, duplicate samples of all five non-sterilized soils (2 g, dry weight equivalent) were pre-equilibrated with a 0.01 M $CaCl_2$ solution and treated by adding 7.4 mL of a 0.01 M $CaCl_2$ solution (1:3.75 ratio) containing [^{14}C]iodomethane to non-silanized glass tubes (p. 25). An additional set of duplicate samples of sterilized California sandy loam soil was prepared and treated as described. The nominal concentrations for the non-sterilized and sterilized soils were approximately 3 and 0.1 µg/mL, respectively. The samples were shaken on a reciprocal shaker overnight at 20°C for 24 hours, then centrifuged at room temperature (pp. 25-26). Triplicate aliquots (0.1 mL) were analyzed for total radioactivity using LSC. The adsorption equilibrium time was determined based on the percent adsorption of the test substance to each test soil. To determine the stability of [^{14}C]iodomethane, aliquots (0.25 or 1 mL) of the supernatants from the 0.1 and 3 µg/mL samples were analyzed using HPLC (p. 26).

Following adsorption, the desorption phase of the preliminary experiment was conducted by replacing the adsorption solution with an equivalent volume of pesticide-free 0.01 M $CaCl_2$ solution and equilibrating on a reciprocal shaker for 24 hours at 20°C (p. 26). The samples were then centrifuged at room temperature. Triplicate aliquots (0.1-0.5 mL) were analyzed for total radioactivity using LSC. Desorption parameters were determined based on the percent desorption of the test substance to each soil. To determine the stability of [^{14}C]iodomethane in the desorption soil:$CaCl_2$ supernatant solution at 24 hours, aliquots of the supernatants from German sandy loam soil and loam soil treated at 3 µg/mL were analyzed using HPLC. To determine the mass balance, single soil samples were combusted and analyzed by LSC. The second set of soil samples were extracted by shaking with 0.1 N sodium hydroxide at 20°C. The samples were centrifuged at room temperature and triplicate aliquots (0.2-mL) were analyzed using LSC.

A final preliminary study was conducted to determine equilibration time of irradiated, sterilized soil (≥26 kGy; p. 27). To determine the equilibration time to be used in the definitive study, samples of each of the five of sterilized soils (2.5 g, dry weight equivalent) were added with a 0.01 M $CaCl_2$ solution to non-silanized glass test tubes and pre-equilibrated overnight on a reciprocal shaker the day before the experiment. Following pre-equilibration, the samples were treated with 80 µL of a 0.01 M $CaCl_2$ solution containing [^{14}C]iodomethane at a nominal concentration of approximately 3 µg/mL. The samples were shaken on a reciprocal shaker at 20°C. Samples were removed after 2, 6, 12, 24, 48, and 72 hours of shaking. The samples were then centrifuged at room temperature, and triplicate 0.1-mL aliquots were analyzed for total radioactivity using LSC. To determine the stability of [^{14}C]iodomethane, single aliquots (0.25 or 1 mL) of the 24-hour supernatant samples were analyzed using HPLC.

Based on the results of the preliminary studies, a soil:solution ratio of 1:3 and an equilibration period of 24 hours were selected for use in the definitive study (pp. 37 and 39). The results of the preliminary studies also determined that sterilized soils and minimal head space (100 µL) were needed in the definitive test to minimize microbial degradation and volatilization (p. 40). It was determined that iodomethane does not adsorb to glass vessels.

2. Definitive study experimental conditions:

Table 3: Study design for the adsorption phase.

Parameters		Swiss loam	CA sandy loam	OH clay loam	German sandy loam	OR silt loam
Condition of soil (air dried/fresh)		Air-dried	Air-dried	Air-dried	Air-dried	Air-dried
Have these soils been used for other laboratory studies ?		No	Yes, aerobic soil metabolism	No	No	No
Soil (g/replicate)		2.5	2.5	2.5	2.5	2.5
Equilibrium solution used (name and concentration		0.01 M $CaCl_2$	0.01 M $CaCl_2$	0.01 M $CaCl_2$	0.01 M $CaCl_2$	0.01 M $CaCl_2$
Control used (with salt solution only) (Yes/No)		Yes	Yes	Yes	Yes	Yes
Test material concentrations	Nominal application rates (mg a.i./kg)*	9.05, 2.92, 0.88, 0.29	9.05, 2.92, 0.88, 0.29	9.05, 2.92, 0.88, 0.29	9.05, 2.92, 0.88, 0.29	9.05, 2.92, 0.88, 0.29
	Analytically measured concentrations (mg/kg; Tables 8-12, pp. 52-56)*	9.28, 3.15, 0.84, 0.29	9.26, 3.07, 0.88, 0.29	9.37, 3.12, 0.85, 0.29	9.23, 3.18, 0.82, 0.29	9.37, 3.18, 0.76, 0.26
Identity and concentration of co-solvent, if any		None	None	None	None	None
Soil:solution ratio		1:3	1:3	1:3	1:3	1:3
Initial pH of the equilibration solution, if provided		Not provided	Not provided	Not provided	Not provided	Not provided

Parameters		Swiss loam	CA sandy loam	OH clay loam	German sandy loam	OR silt loam
No. of replications	Controls	2	2	2	2	2
	Treatments	2	2	2	2	2
Equilibration	Time (hours)	24	24	24	24	24
	Temperature (°C)	20	20	20	20	20
	Darkness (Yes/No)	Not provided	Not provided	Not provided	Not provided	Not provided
	Shaking method	Mechanical	Mechanical	Mechanical	Mechanical	Mechanical
	Shaking time (hours)	24	24	24	24	24
Method of separation of supernatant (eg., centrifugation)		Centrifugation	Centrifugation	Centrifugation	Centrifugation	Centrifugation
Centrifugation	Speed (rpm)	Not provided	Not provided	Not provided	Not provided	Not provided
	Duration (min)	Not provided	Not provided	Not provided	Not provided	Not provided
	Method of separation of soil and solution	Decantation	Decantation	Decantation	Decantation	Decantation

Data were obtained from pp. 28-29 of the study report.

* Reviewer-calculated by multiplying the concentration of the test substance by the amount of $CaCl_2$ solution divided by the amount of soil, *e.g.*, 3.1 x 7.3 ÷ 2.5 = 9.1.

Table 4: Study design for the desorption phase.

Parameters		Swiss loam	CA sandy loam	OH clay loam	German sandy loam	OR silt loam
Were the soil residues from the adsorption phase used? If not, describe the method for adsorption using a separate adsorption Table		Yes	Yes	Yes	Yes	Yes
Amount of test material present in the adsorbed state/ adsorbed amount (mg a.i./kg soil)*	9.05	1.4120	1.6266	2.4381	1.0511	1.8363
	2.9	0.4438	0.4315	1.0096	0.3311	0.7033
	0.88	0.1190	0.1754	0.2307	0.0771	0.1782
	0.29	0.0443	0.0558	0.0845	0.0428	0.0672
No. of desorption cycles		1	1	1	1	1
Equilibration solution and quantity used per treatment for desorption (eg., 0.01 M $CaCl_2$)		0.01 M $CaCl_2$	0.01 M $CaCl_2$	0.01 M $CaCl_2$	0.01 M $CaCl_2$	0.01 M $CaCl_2$
Soil:solution ratio		1:3	1:3	1:3	1:3	1:3
Replications	Controls	2	2	2	2	2
	Treatments	2	2	2	2	2
Desorption equilibration	Time (hours)	24	24	24	24	24
	Temperature (°C)	20	20	20	20	20
	Darkness	Not provided	Not provided	Not provided	Not provided	Not provided
	Shaking method	Mechanical	Mechanical	Mechanical	Mechanical	Mechanical
	Shaking time (hours)	24	24	24	24	24

Parameters		**Swiss loam**	**CA sandy loam**	**OH clay loam**	**German sandy loam**	**OR silt loam**
Centrifugation	Speed (rpm or g)	Not provided	Not provided	Not provided	Not provided	Not provided
	Duration (min)	Not provided	Not provided	Not provided	Not provided	Not provided
	Method of separation of soil and solution	Decantation	Decantation	Decantation	Decantation	Decantation
Second desorption	Indicate if the method is same as the first desorption cycle.	Not performed				

Data were obtained from p. 29 and Tables 8-12, pp. 52-56 of the study report.
* Means were reviewer-calculated using Excel.

3. Description of analytical procedures:

Extraction/clean up/concentration methods: No extractions were performed.

Total ^{14}C measurement: Triplicate aliquots (0.1 mL) of the supernatants were analyzed for total radioactivity using LSC (p. 29). Following desorption, soil residues were combusted then analyzed by LSC (p. 31). Combustion efficiency was >95%; radioactive recoveries in the subsamples were corrected for oxidizer efficiency.

Non-extractable residues, if any: Not applicable.

Derivatization method, if used: A derivatization method was not employed in the study.

Identification and quantification of parent compound: The aqueous dosing solution and supernatant samples were analyzed by HPLC (p. 30). Identification and quantification of the parent compound were performed by HPLC using the following operating conditions: BioRad Aminex HPX-87H column (7.8 mm x 30 cm), isocratic gradient mobile phase using (A) aqueous sulfuric acid (0.005M), flow rate 1.0 mL/minute, and UV (220 nm) and radioactive monitoring detection. The identity of the test material was confirmed by HPLC co-injection of [^{14}C]iodomethane with an authentic standard of iodomethane and by MS (p. 37). The identity of iodomethane in the supernatant was confirmed by HPLC cochromatography with the dosing solution (p. 43; Figure 19, p. 77).

Identification and quantification of transformation products: Identification and quantification of transformation products were not performed.

Detection limits (LOD, LOQ) for the parent compound: The detection limit (LOD) for LSC analyses was reported as twice background (p. 32). The LOQ for LSC analyses was <0.001 ppm for a 1 mL sample size. Detection limits (LOD and LOQ) for HPLC analyses of the parent compound were not reported.

Detection limits (LOD, LOQ) for the transformation products: Identification and quantification of transformation products were not performed.

II. RESULTS AND DISCUSSION

A. TEST CONDITIONS: The study was conducted according to the protocol with the exception of the protocol amendments included in this report. It was not stated whether the equilibration periods were conducted in the dark. The temperature during the equilibration periods was reported to be maintained at 20°C; however, temperature data were not provided. HPLC analysis of the adsorption supernatants showed that >96% of the radioactivity was parent compound (p. 43; Figures 13-17, pp. 71-75).

B. MASS BALANCE: The mass balance was not reported at the end of adsorption phase of the study. Mass balances were calculated by summing the total amount of iodomethane recovered in the adsorption and desorption solutions and soil residues (p. 43). The complete mass balance for the high-dose soils (adsorption and desorption) was 87.0-88.3%, 84.4-87.6%, 86.4-89.8%, 91.4-93.9%, and 86.7-90.4% of the applied for the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively (Table 14, p. 58).

Table 5: Recovery of iodomethane, expressed as percentage of applied radioactivity, in soil after adsorption/desorption (n = 8, mean ± s.d.).*

Matrices	Loam	California sandy loam	Clay loam	German sandy loam	Silt loam
At the end of the adsorption phase					
Supernatant solution[1]	84.4 ± 1.7	81.4 ± 4.7	70.8 ± 2.6	87.5 ± 2.6	76.6 ± 2.7
Solid phase (total ^{14}C)[2]	14.4 ± 1.7	17.4 ± 4.7	28.2 ± 2.7	11.3 ± 2.6	22.3 ± 2.8
Non-extractable residues in soil	Not determined				
Total recovery following adsorption	Not determined				
At the end of the desorption phase					
Supernatant solution[1]	17.6 ± 0.4	14.0 ± 0.8	23.7 ± 0.9	15.9 ± 1.0	19.9 ± 1.0
Solid phase (total ^{14}C)[2]	15.3 ± 2.0	15.3 ± 4.3	23.4 ± 3.0	11.1 ± 1.8	17.4 ± 1.7
Non-extractable residues in soil, if measured	Not determined				
Total recovery[3]	87.5 ± 0.7	86.0 ± 2.3	88.1 ± 2.4	92.6 ± 1.8	88.6 ± 2.6

* Means and standard deviations were calculated by the reviewer using Excel and data obtained from Tables 8-12, pp. 52-56 and Table 14, p. 58 in the study report.

[1] The percent in the supernatant solution was reviewer calculated by dividing the amount present by the amount applied x 100, *e.g.*, 2.6903 x 7.3 ÷ 23.53 x 100 = 83.5%.

[2] The percent in the solid phase was reviewer-calculated by dividing the amount present by the amount applied x 100, *e.g.*, 1.4487 x 2.5 ÷ 23.53 x 100 = 15.4%.

[3] The total recovery was determined by the study authors for the high-dose soils only.

Table 6: Concentration of iodomethane in the solid and liquid phases at the end of adsorption equilibration period (n = 2, mean ± s.d.).*

Concentration (mg a.i/kg)	Loam			California sandy loam			Clay loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]
9.05	1.4120± 0.1	2.7027 ± 0.0	15.0 ± 0.6	1.6266 ± 0.1	2.6208 ± 0.1	17.3 ± 1.6	2.4381 ± 0.0	2.3858± 0.0	25.7 ± 0.2
2.9	0.4438 ± 0.0	0.9298 ± 0.0	13.9 ± 0.6	0.4315 ± 0.1	0.9043 ± 0.0	13.9 ± 2.0	1.0096 ± 0.1	0.7292 ±0.0	31.9 ± 2.0
0.88	0.1190 ± 0.0	0.2504 ± 0.0	13.8 ± 0.8	0.1754 ± 0.1	0.2408 ± 0.0	19.8±10.4	0.2307 ± 0.0	0.2126 ±0.0	26.8 ± 0.6
0.29	0.0443 ± 0.0	0.0851 ± 0.0	15.0 ± 4.2	0.0558 ± 0.0	0.0812 ± 0.0	18.8 ± 1.6	0.0845 ± 0.0	0.0715 ±0.0	28.6 ± 1.2

Concentration (mg a.i/kg)	German sandy loam			Silt loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]
9.05	1.0511 ± 0.1	2.8044 ± 0.0	11.2 ± 1.5	1.8363 ± 0.1	2.5891 ± 0.0	19.3± 1.2
2.9	0.3311 ± 0.0	0.9787 ± 0.0	10.3 ± 1.2	0.7033 ± 0.1	0.8530 ± 0.0	21.8 ± 2.4
0.88	0.0771 ± 0.0	0.2537 ± 0.0	9.3 ± 1.7	0.1782 ± 0.0	0.1993 ± 0.0	23.2 ± 3.0
0.29	0.0428 ± 0.0	0.0856 ± 0.0	14.4 ± 3.2	0.0672 ± 0.0	0.0679 ± 0.0	25.1 ± 1.4

*Means and standard deviations were calculated by the reviewer using Excel and data obtained from Tables 8-12, pp. 52-56 of the study report.

[1] The % adsorbed was obtained by dividing the amount of iodomethane on the soil by the amount applied, e.g. 1.4487 x 2.5 ÷ 21.53 x 100 = 16.8%.

Table 7: Concentration of iodomethane in the solid and liquid phases at the end of desorption (n = 2, total of all desorptions).*

Concentration (mg a.i./kg)	Loam			California sandy loam			Clay loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1,2]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1]
9.05	1.4647	0.5520	0	1.4411	0.4171	11.6	2.0180	0.7737	17.2
2.9	0.4824	0.1952	0	0.3590	0.1611	16.8	0.8699	0.2470	13.8
0.88	0.1216	0.0528	0	0.1518	0.0429	13.3	0.1825	0.0711	20.9
0.29	0.0487	0.0180	0	0.0510	0.0141	8.7	0.0701	0.0245	17.2

Concentration (mg a.i./kg)	German sandy loam			Silt loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1,2]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1]
9.05	1.0712	0.4823	0	1.5543	0.6057	15.6
2.9	0.3528	0.1746	0	0.5611	0.2211	20.2
0.88	0.0755	0.0460	1.9	0.1337	0.0551	25.0
0.29	0.0382	0.0169	9.2	0.0546	0.0184	18.8

* Means were reviewer-calculated using Excel and data obtained from Tables 8-12, pp. 52-56 of the study report.

[1] The % desorbed as % of the adsorbed was calculated for each sample as follows: 100 - (the amount on soil after desorption divided by the amount after adsorption x 100), *e.g.*, 100 - (1.5807 ÷ 1.7312 x 100) = 8.7%.

[2] The % desorbed as % of the adsorbed was reported as 0% if the calculation yielded a negative number, indicating no desorption of the test substance to the test soil occurred.

Table 8: Adsorption and desorption constants of iodomethane in the soils.

Soil	Adsorption				Desorption			
	K	1/N	R^2	K_{oc}	K_d	1/N	R^2	K_{oc}
Loam	0.5	1.0049	0.9905	28	2.5	1.0051	0.9912	142
California sandy loam	0.6	0.9328	0.9501	59	2.7	0.9417	0.9466	265
Clay loam	1.1	0.9836	0.9916	26	2.9	1.0041	0.9875	67
German sandy loam	0.4	0.9391	0.9713	15	2.1	1.0131	0.9843	77
Silt loam	0.8	0.9126	0.9953	41	2.4	0.9666	0.9927	123

Data were obtained from Table 13, p. 57 of the study report.
K - Freundlich adsorption coefficients; 1/N - Slope of Freundlich adsorption isotherms.
K_{oc} - Coefficient adsorption per organic carbon (K_d or K x 100/% organic carbon).
R^2 - Regression coefficient of Freundlich equation.

C. ADSORPTION: After 24 hours of equilibration, 13.8-15.0%, 13.9-19.8%, 25.7-31.9%, 9.3-14.4%, and 19.3-25.1% of the applied iodomethane was adsorbed to the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively (reviewer-calculated). Adsorption K_d values were 0.5, 0.6, 1.1, 0.4, and 0.8 mL/g for the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively (Table 13, p. 57). Adsorption K_{oc} values were 28, 59, 26, 15, and 41 mL/g for the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively.

D. DESORPTION: At the end of desorption, 0%, 8.7-16.8%, 13.8-20.9%, 0-.9.2%, and 15.6-25.0% of the applied ^{14}C was desorbed in the loam, California sandy loam, clay loam, German sandy loam and silt loam soils, respectively (reviewer-calculated). Freundlich K_{des} values were 2.5, 2.7, 2.9, 2.1, and 2.4 mL/g for the loam, California sandy loam, clay loam, German sandy loam, and silt loam, respectively (Table 13, p. 57). Desorption K_{oc} values were 142, 265, 67, 77, and 123 mL/g for the loam, California sandy loam, clay loam, German sandy loam, and silt loam soils, respectively.

III. STUDY DEFICIENCIES: The objective of this study was to determine the adsorption and desorption properties of [^{14}C]iodomethane in soils. None of the study deficiencies noted are considered to be of sufficient concern to cause the study to be judged scientifically invalid. However, since none of the test soils had an organic matter content ≤1%, this study cannot be used to fulfill Subdivision N Guideline §163-1. This study does provide useful supplemental information on the mobility of iodomethane in five soils.

IV. REVIEWER'S COMMENTS:

1. Material balances were outside the acceptable guideline range of 90 to 110% of the applied radioactivity for both replicates of the high-dose loam (87.0-88.3% of the applied), California sandy loam (84.4-87.6% of the applied), and clay loam (86.4-89.8% of the applied) soils, and for one of two replicates of the high-dose silt loam soil (86.7% of the applied; Table 14, p. 58). Both high-dose replicates of the German sandy loam had acceptable material balances (91.4-93.9% of the applied), as did the remaining high-dose silt loam replicate (90.4% of the applied). The study authors acknowledged the low material balances and stated that some losses of the test material could not be avoided during sampling handling due to iodomethane's extreme volatility (p. 43). This explanation is supported by the results obtained in aerobic soil metabolism and terrestrial field dissipation studies which demonstrated that volatilization is the major route of dissipation of iodomethane (MRIDs 45593707 and 45593711; reviews included in this submission).

2. Complete details of the experimental design were not provided. It was not stated whether the adsorption and desorption phases of the experiment were conducted in the dark. Subdivision N guidelines specify that mobility studies should be conducted in the dark to minimize photodegradation of the test substance. Based on the results of a photolysis study, some degradation occurred in irradiated samples between 0 and 1 day posttreatment (MRID 45593706; review included in this submission). Iodomethane decreased from 99.33% of the applied at time 0 to 94.92% of the applied in the irradiated samples compared to 98.33% of the applied in the dark controls at 1 day posttreatment. In the present mobility study, if the 24-hour equilibration periods in the present study were not conducted in darkness, then some degradation of iodomethane would be expected to occur, based on the findings in the photolysis study. The registrant should specify whether the definitive study was conducted in the dark.

   In addition, the temperature during the definitive study was reported to be 20°C; however, temperature records were not provided in the study.

3. Sterilization of the test soils used in the definitive test was achieved using gamma irradiation. This method is considered acceptable since the physical and chemical properties of soil are not altered. Sterilization of the test soils used in the preliminary kinetics tests was achieved by autoclaving the soils for 30 minutes at 121°C (p. 24). The study authors did not explain why autoclaving was selected as the method for soil sterilization in the preliminary experiments. Autoclaving changes the physical and chemical properties of the soils, and therefore, is not considered acceptable.

4. The loam soil and one sandy loam soil were foreign in origin, but were characterized according to the USDA soil textural classification system and were comparable to soils found in the United States.

5. The California sandy loam soil was the same soil used in the aerobic soil metabolism study, which was reviewed in this submission (pp. 20 and 44).

6. It was stated that the samples were stored refrigerated at 4°C, but the duration of sample storage was not specified (p. 31).

7. Method detection limits for HPLC analyses were not reported. Both method detection limits and limits of quantification should be reported to allow the reviewer to evaluate the adequacy of the analytical method.

8. The maximum proposed field application rate for iodomethane was not reported. Subdivision N guidelines specify that for batch equilibrium studies, one concentration of the test substance should be roughly equivalent to the maximum proposed or registered field application rate of the parent compound.

V. REFERENCES: The following references were cited in the study:

Soil quality – Sampling – Part 6: Guidance on the collection, handling and storage of soil for the assessment of aerobic microbial processes in the laboratory, ISO 10386-6:1993(E), International Organization for Standardization Genève, 1993.

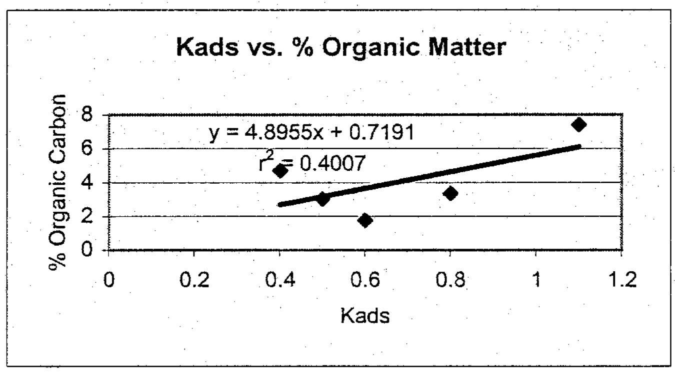
Wujcik, C.J. 2001. "An Aerobic Soil Metabolism Study with [^{14}C]Iodomethane," Ricerca Study Number 012131.

Attachment 1

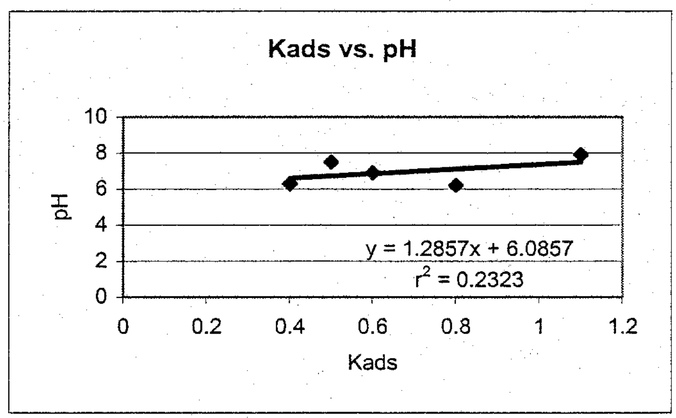
Excel Spreadsheets

Chemical Name	Iodomethane
PC Code	000011
MRID	45593709
Guideline No.	163-1

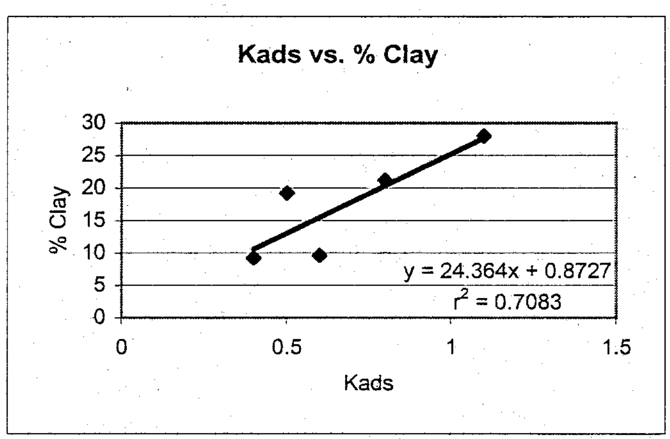
Soil	Kads	% Organic Matter
Loam	0.5	3.03
Sandy loam	0.6	1.76
Clay loam	1.1	7.4
Sandy loam	0.4	4.7
Silt loam	0.8	3.35



Soil	Kads	pH
Loam	0.5	7.5
Sandy loam	0.6	6.9
Clay loam	1.1	7.9
Sandy loam	0.4	6.3
Silt loam	0.8	6.2



Soil	Kads	% Clay
Loam	0.5	19.2
Sandy loam	0.6	9.6
Clay loam	1.1	28
Sandy loam	0.4	9.2
Silt loam	0.8	21.2



Chemical Name: Iodomethane
163-1
MRID 45593709

Table 4 and 6 Soil adosrption	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
3.0	1.4487	1.7312	2.4549	1.1504	1.9194
3.0	1.3753	1.5220	2.4212	0.9518	1.7531
Average	1.4120	1.6266	2.4381	1.0511	1.8363
STDEV	0.0519	0.1479	0.0238	0.1404	0.1176
1.0	0.4311	0.4763	1.0548	0.3044	0.7576
1.0	0.4565	0.3866	0.9643	0.3577	0.6490
Average	0.4438	0.4315	1.0096	0.3311	0.7033
STDEV	0.0180	0.0634	0.0640	0.0377	0.0768
0.3	0.1144	0.1101	0.2272	0.0670	0.1618
0.3	0.1236	0.2406	0.2342	0.0871	0.1946
Average	0.1190	0.1754	0.2307	0.0771	0.1782
STDEV	0.0065	0.0923	0.0049	0.0142	0.0232
0.1	0.0355	0.0592	0.0870	0.0361	0.0646
0.1	0.0530	0.0524	0.0820	0.0494	0.0697
Average	0.0443	0.0558	0.0845	0.0428	0.0672
STDEV	0.0124	0.0048	0.0035	0.0094	0.0036

Table 5 % in Supernatant	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
3.0	83.46	80.45	73.16	86.51	78.72
3.0	84.23	82.65	73.51	88.61	80.44
1.0	85.34	83.53	65.80	89.35	75.50
1.0	84.56	86.38	68.62	87.72	78.82
0.3	85.53	86.42	72.59	90.67	77.87
0.3	84.48	71.91	71.78	88.27	73.65
0.1	86.81	78.92	69.65	86.61	74.85
0.1	80.99	81.19	71.32	82.17	73.00
Average	84.43	81.43	70.80	87.49	76.60
s.d.	1.71	4.68	2.63	2.55	2.72

Table 5 % on Soil	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
3.0	15.39	18.45	25.84	12.30	20.20
3.0	14.61	16.22	25.49	10.18	18.45
1.0	13.49	15.32	33.30	9.43	23.47
1.0	14.28	12.44	30.44	11.08	20.11
0.3	13.30	12.40	26.42	8.09	21.07
0.3	14.37	27.09	27.23	10.52	25.34
0.1	11.99	20.00	29.39	12.20	24.10
0.1	17.91	17.70	27.70	16.69	26.01
Average	14.42	17.45	28.23	11.31	22.34
s.d.	1.74	4.74	2.66	2.58	2.75

Table 5	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
% des. Supernatant					
3.0	17.17	13.03	23.29	14.76	18.05
3.0	17.08	12.92	24.27	15.36	19.18
1.0	18.19	14.82	22.72	15.74	19.90
1.0	17.48	15.45	22.81	15.85	20.10
0.3	17.96	14.24	24.07	16.33	20.80
0.3	17.89	13.65	24.21	16.08	21.10
0.1	17.76	13.71	22.89	15.19	19.94
0.1	17.66	14.01	25.35	18.05	20.16
Average	17.65	13.98	23.70	15.92	19.90
s.d.	0.39	0.85	0.93	1.00	0.95

Table 5	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
Solid phase					
3.0	16.21	16.84	20.80	12.86	18.06
3.0	14.91	13.87	21.68	10.04	14.66
1.0	14.34	12.73	28.34	10.17	18.79
1.0	15.84	10.37	26.57	11.68	15.97
0.3	13.59	10.80	20.84	8.07	15.94
0.3	14.67	23.38	21.60	10.16	18.87
0.1	13.31	18.21	25.95	12.20	17.60
0.1	19.59	16.22	21.42	13.61	19.26
Average	15.31	15.30	23.40	11.10	17.39
s.d.	2.00	4.31	3.03	1.81	1.68

Table 5	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
Total Recovery					
3.0	87.00	84.40	89.80	91.40	86.7
3.0	88.00	87.60	86.40	93.90	90.4
average	87.50	86.00	88.10	92.65	88.55
s.d.	0.71	2.26	2.40	1.77	2.62

	Table 6 Solution	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
	3.0	2.6903	2.5854	2.3801	2.7708	2.5610
	3.0	2.7151	2.6561	2.3915	2.8379	2.6172
Average		2.7027	2.6208	2.3858	2.8044	2.5891
STDEV		0.0175	0.0500	0.0081	0.0474	0.0397
	1.0	0.9341	0.8891	0.7139	0.9877	0.8346
	1.0	0.9255	0.9194	0.7445	0.9697	0.8713
Average		0.9298	0.9043	0.7292	0.9787	0.8530
STDEV		0.0061	0.0214	0.0216	0.0127	0.0260
	0.3	0.2519	0.2628	0.2138	0.2571	0.2048
	0.3	0.2488	0.2187	0.2114	0.2503	0.1937
	Average	0.2504	0.2408	0.2126	0.2537	0.1993
	STDEV	0.0022	0.0312	0.0017	0.0048	0.0078
	0.1	0.0880	0.0800	0.0706	0.0878	0.0687
	0.1	0.0821	0.0823	0.0723	0.0833	0.0670
	Average	0.0851	0.0812	0.0715	0.0856	0.0679
	STDEV	0.0042	0.0016	0.0012	0.0032	0.0012

Table 6 % Adsorbed	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
3.0	15.39	18.45	25.84	12.30	20.20
3.0	14.61	16.22	25.49	10.18	18.45
Average	15.00	17.33	25.66	11.24	19.33
STDEV	0.55	1.58	0.25	1.50	1.24
1.0	13.49	15.32	33.30	9.43	23.47
1.0	14.28	12.44	30.44	11.08	20.11
Average	13.89	13.88	31.87	10.26	21.79
STDEV	0.56	2.04	2.02	1.17	2.38
0.3	13.30	12.40	26.42	8.09	21.07
0.3	14.37	27.09	27.23	10.52	25.34
Average	13.84	19.75	26.83	9.31	23.20
STDEV	0.76	10.39	0.58	1.72	3.02
0.1	11.99	20.00	29.39	12.20	24.10
0.1	17.91	17.70	27.70	16.69	26.01
Average	14.95	18.85	28.55	14.44	25.06
STDEV	4.18	1.62	1.19	3.18	1.35

Table 7 On Soil		Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
	3.0	1.5256	1.5807	1.9760	1.2030	1.7156
	3.0	1.4037	1.3014	2.0599	0.9393	1.3929
Average		1.4647	1.4411	2.0180	1.0712	1.5543
STDEV		0.0862	0.1975	0.0593	0.1865	0.2282
	1.0	0.4584	0.3958	0.8979	0.3284	0.6065
	1.0	0.5064	0.3222	0.8418	0.3771	0.5156
Average		0.4824	0.3590	0.8699	0.3528	0.5611
STDEV		0.0339	0.0520	0.0397	0.0344	0.0643
	0.3	0.1169	0.0959	0.1792	0.0668	0.1224
	0.3	0.1262	0.2076	0.1858	0.0841	0.1449
	Average	0.1216	0.1518	0.1825	0.0755	0.1337
	STDEV	0.0066	0.0790	0.0047	0.0122	0.0159
	0.1	0.0394	0.0539	0.0768	0.0361	0.0521
	0.1	0.0580	0.0480	0.0634	0.0403	0.0570
	Average	0.0487	0.0510	0.0701	0.0382	0.0546
	STDEV	0.0132	0.0042	0.0095	0.0030	0.0035

Table 7 Solution		Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
	3.0	0.5533	0.4189	0.7578	0.4728	0.5873
	3.0	0.5506	0.4153	0.7895	0.4918	0.6240
Average		0.5520	0.4171	0.7737	0.4823	0.6057
STDEV		0.0019	0.0025	0.0224	0.0134	0.0260
	1.0	0.1991	0.1577	0.2465	0.1740	0.2200
	1.0	0.1913	0.1644	0.2475	0.1752	0.2222
Average		0.1952	0.1611	0.2470	0.1746	0.2211
STDEV		0.0055	0.0047	0.0007	0.0008	0.0016
	0.3	0.0529	0.0443	0.0709	0.0463	0.0547
	0.3	0.0527	0.0415	0.0713	0.0456	0.0556
	Average	0.0528	0.0429	0.0711	0.0460	0.0551
	STDEV	0.0001	0.0020	0.0003	0.0005	0.0006
	0.1	0.0180	0.0139	0.0232	0.0154	0.0183
	0.1	0.0179	0.0142	0.0257	0.0183	0.0185
	Average	0.0180	0.0141	0.0245	0.0169	0.0184
	STDEV	0.0001	0.0002	0.0018	0.0021	0.0001

Table 7	Swiss Loam	CA Sandy loam	OH Clay loam	Ger. Sandy loam	OR Silt loam
% Desorbed					
3.0	-5.31	8.69	19.51	-4.57	10.62
3.0	-2.07	14.49	14.92	1.31	20.55
Average	-3.69	11.59	17.22	-1.63	15.58
STDEV	2.29	4.10	3.24	4.16	7.02
1.0	-6.33	16.90	14.87	-7.88	19.94
1.0	-10.93	16.66	12.70	-5.42	20.55
Average	-8.63	16.78	13.79	-6.65	20.25
STDEV	3.25	0.17	1.54	1.74	0.43
0.3	-2.19	12.90	21.13	0.30	24.35
0.3	-2.10	13.72	20.67	3.44	25.54
Average	-2.14	13.31	20.90	1.87	24.95
STDEV	0.06	0.58	0.33	2.22	0.84
0.1	-10.99	8.95	11.72	0.00	19.35
0.1	-9.43	8.40	22.68	18.42	18.22
Average	-10.21	8.67	17.20	9.21	18.79
STDEV	1.10	0.39	7.75	13.03	0.80